

Role of growth temperature on formation of single crystalline GaN nanorods on flexible titanium foil by laser molecular beam epitaxy

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ABSTRACT

We have studied the effect of growth temperature (550–700 °C) on the morphological, structural and optical properties of GaN nanostructures grown directly on flexible Ti metal foil by laser assisted molecular beam epitaxy (LMBE). The low growth temperature (550 °C) favors a growth of dense, three-dimensional GaN islands whereas probe-shaped sparse GaN nanorods (NRs) are obtained at 700 °C. The average length, diameter and density of GaN NRs were analyzed from field emission scanning electron microscopy images and were estimated to be 80 nm, 260 nm and $\sim 9.6 \times 10^8 \text{ cm}^{-2}$ respectively. The high-resolution transmission electron microscopy analysis confirmed that the GaN NRs have single crystalline structure over the entire length and had grown along c-axis. Raman spectroscopy studies revealed that the LMBE grown GaN nanostructures on Ti foil possess wurtzite structure with a low tensile stress (0.15–0.37 GPa). The intense near band edge emission peak appeared at 3.42 eV, similar to bulk GaN, with a smaller full width at half maximum of 100 meV for the sparse GaN NRs grown at 700 °C. Direct growth of GaN NRs at a relatively lower temperature on flexible metal foils with high structural and optical quality holds the promise for the fabrication of flexible GaN based futuristic optoelectronics devices.

1. Introduction

One dimensional (1D) GaN nanostructures such as nanowires (NWs), nanorods (NRs) and nanotubes (NTs) are attractive for various nanoscale device applications due to their exciting exotic physical and optical properties [1–3]. Various 1D GaN nanostructures have been grown mostly on the conventional substrates such as sapphire and silicon using different growth techniques [2–9]. Sapphire substrate is an electrical insulator and a poor heat dissipater whereas silicon substrate is high optical absorbent [10]. These properties may affect the performance of GaN based light emitting diodes (LEDs) and laser diodes (LDs) [10]. The large aspect and surface-to-volume ratios favor the growth of nearly threading dislocation (TD) free GaN NRs in the upper part since there is expulsion of TDs at the sides of NRs as the growth proceeds [11,12]. Consequently, the growth of GaN NRs is not limited by the choice of lattice-matched substrates. Alternatively, single crystalline metals, metal-coated substrates and flexible metal foils are the interesting options for developing GaN NR based LED and LD devices due to high optical reflection and excellent thermal and electrical properties [10–13]. Also, the flexible metal foils possess advantage over metal substrates due to low cost and flexible nature.

Recently, a couple of research groups have reported the growth of dense GaN NWs on flexible metal foils such as Ti and Ta using plasma assisted molecular beam epitaxy (PA-MBE) [11–13]. May et al. have reported the growth of dense GaN NWs on Ti and Ta metal foils at 800 °C using PAMBE technique in N-rich growth condition [11]. Calabrese et al. also reported the growth of grass-like dense GaN NWs on flexible Ti metal foil using PAMBE at the growth temperature of 730 °C [12,13]. The growth of sparse GaN NRs with preferred orientation and regular arrays is preferred over random oriented grown NRs for various application perspectives [14]. Additionally, the growth of GaN on metal foils at high growth temperature may form some unwanted interfacial alloys or compounds due to the reactive nature of metals which may affect the structural, electronic and optical properties of the NRs. Laser assisted MBE (LMBE) is considered as the relatively lower growth temperature technique since high kinetically-energized precursors produced by laser ablation of GaN target reduce the growth temperature. However, as per available literature, there is no report on the LMBE growth and characterization of sparse GaN NRs on flexible Ti metal foil.

In the current work, we report the effect of growth temperature on the formation of self-assembled GaN NR on Ti foil using LMBE system

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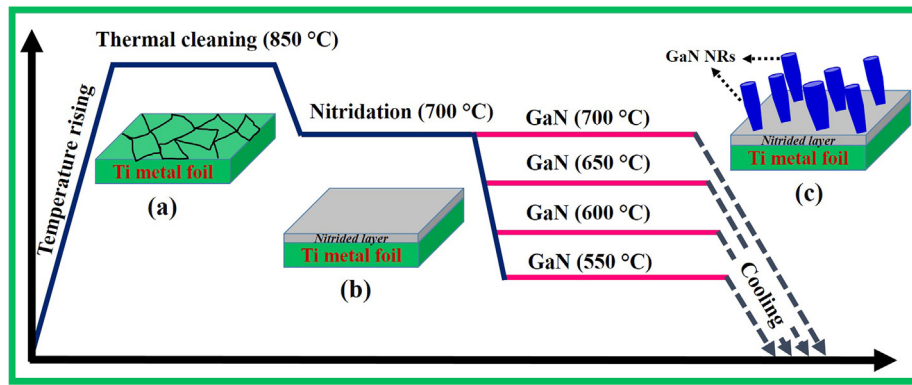


Fig. 1. The schematic diagram of GaN growth sequence on flexible Ti metal foil using LMBE technique. (a) Thermal cleaning of Ti metal foil at 850 °C; (b) Nitridation of Ti foil at 700 °C; (c) growth of GaN nanorods at growth temperature of 700 °C on nitridated Ti metal foil.

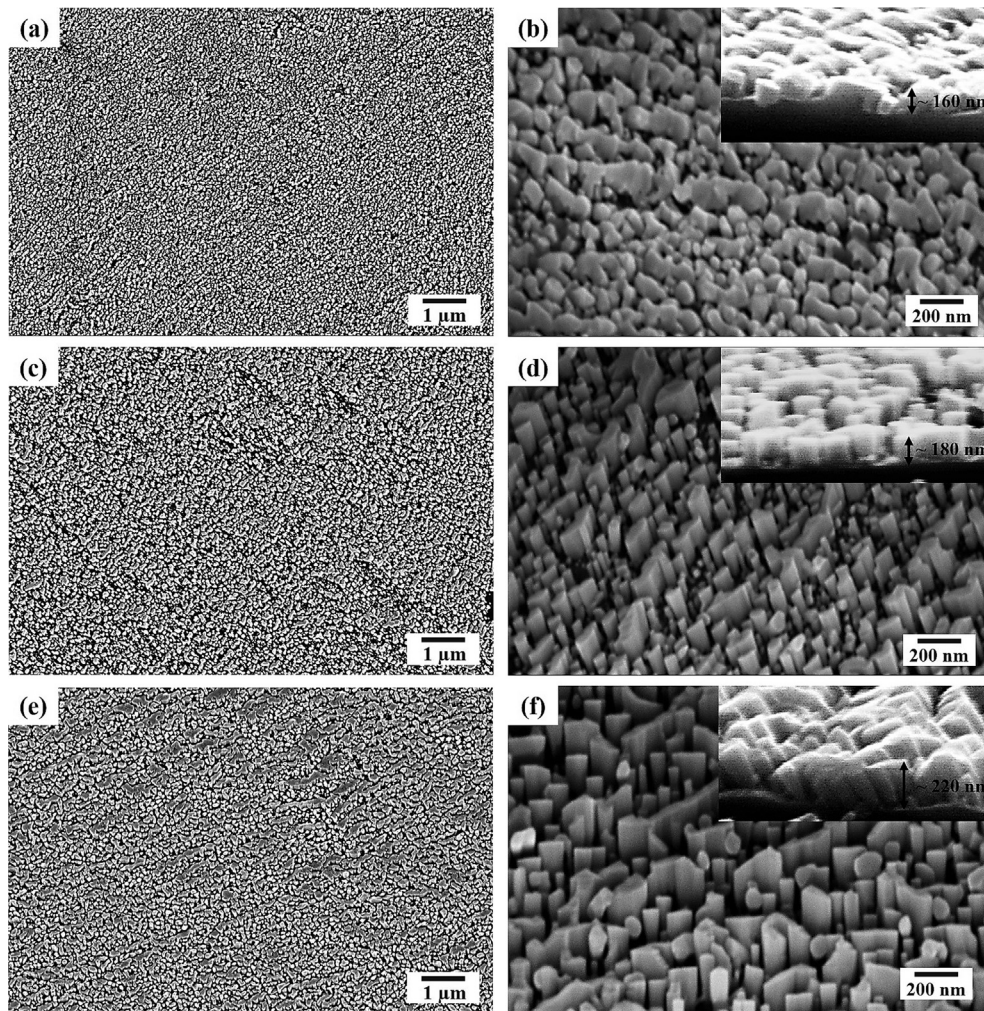


Fig. 2. Plan and 45° tilt-view FE-SEM images of LMBE grown GaN nanostructures on nitridated Ti flexible metal foil at different growth temperatures of: (a and b) 550, (c and d) 600 and (e and f) 650 °C. Insets of (b, d and f) show the cross-sectional view FE-SEM images for GaN growth at 550, 600 and 650 °C, respectively.

over a wider window, i.e. 550–700 °C that has not been studied before. The sparse single crystalline GaN NRs grown along c-axis are obtained at 700 °C and the NRs have a wurtzite hexagonal structure. They exhibit a sharp and intense band-to-band emission at 3.42 eV with negligible deep bands at room temperature. The GaN NRs on Ti foil can serve as template for developing III-nitride based flexible electronic and optoelectronic devices.

2. Experimental

The GaN NRs were grown on flexible nitridated Ti metal foils using ultra-high vacuum (UHV) (2×10^{-10} Torr) LMBE growth technique. The effect of growth temperature in the range of 550–700 °C has been studied. The LMBE system is equipped with various targets, reflection high energy electron diffraction system, r.f. nitrogen plasma cell and residual gas analyzer. The schematic of growth sequence and other

processes is shown in Fig. 1. The Ti metal foil (Alfa Aesar, Thickness ~ 0.127 mm, purity $\sim 99.99\%$) was cleaned with standard organic solvents and then degassed at 250°C for few hours in the load lock chamber. Further, it was thermally cleaned at 850°C for 30 min under UHV condition in the growth chamber. The thermally cleaned Ti metal foil was then nitrided at 700°C by impinging active nitrogen species using r.f. nitrogen plasma with a flow rate of 1.1 sccm semiconductor grade nitrogen gas at an r.f. forward power of 400 W. The GaN was grown on nitridated Ti foil by ablating a hydride vapor phase epitaxy grown polycrystalline GaN solid target (purity: 99.9999%) with KrF excimer laser (wavelength = 248 nm, pulse width = 25 ns) at a fixed laser repetition rate of 20 Hz and an energy density of $\sim 3\text{ J/cm}^2$. Additional nitrogen plasma species were supplied with a nitrogen flow rate of 0.4 sccm and an applied r.f. power of 250 W during GaN growth to maintain an N-rich GaN growth condition.

The surface morphology of LMBE grown GaN samples was examined using field emission scanning electron microscopy (FESEM) with an operating voltage of 5 kV. The x-ray diffraction (XRD) characterization was performed using $\text{Cu K}\alpha_1$ source with the wavelength of 0.15406 nm. The structural analysis and the nature of stress of GaN samples were examined by Raman spectroscopy in backscattered geometry using a 514.5 nm excitation laser source. The high resolution transmission electron microscopy (HR-TEM) characterization was performed on the GaN NRs dispersed on a carbon lacey Cu grid with an operating voltage of 300 kV. The photoluminescence (PL) data were recorded by using Edinburgh Fluorescence spectrometer. A He–Cd laser of 325 nm wavelength was used as the excitation source. The emission arm was equipped with a standard photo-multiplier tube detector.

3. Results and discussion

The plan- and 45° tilt-view FESEM images of GaN grown on nitridated Ti metal foil at growth temperatures in the range of 550 – 650°C are shown in Fig. 2. It is observed that three dimensional (3D) GaN islands have been grown on Ti foil at the growth temperature of 550°C as seen in Fig. 2(a and b). Inset of Fig. 2(b) represents the cross-sectional image and the vertical height of GaN islands estimated to be ~ 160 nm. The statistical analysis showed that these GaN islands possess broad lateral grains of size in range of 40–90 nm. In addition, the prominent coalescence of GaN islands on the steps or grain boundaries of Ti foil was observed as these are the preferred nucleation sites [Fig. 2(a and b)]. The GaN surface morphology changed from island shape to well aligned GaN NR structure when the growth temperature was raised to 600°C as shown in Fig. 2(c and d). The grown GaN NRs are in probe-shape with hexagonal top facets and are having a typical length of ~ 180 nm [inset of Fig. 2(d)]. The coalescence of GaN NRs increased for the GaN grown at 650°C as shown in Fig. 2(e and f). Here, we obtained two distinct GaN features: long individual GaN NRs of ~ 220 nm length estimated by cross-sectional image in Fig. 2(f) and coalesced GaN NRs in the form of boundary wall. The statistical analysis revealed that the densities of the GaN NRs grown at 600 and 650°C are $\sim 1.1 \times 10^{10}$ and $\sim 6.2 \times 10^9\text{ cm}^{-2}$, respectively.

Fig. 3 presents the plan- and 45° tilt-view FESEM images of LMBE grown GaN on nitridated Ti foil at 700°C . Surprisingly, the sparse and tilted individual GaN NRs with negligible coalescence obtained on Ti foil [Fig. 3(b–d)]. May et al. observed various degree of orientation of GaN NWs on polycrystalline Ti foil and suggested an epitaxial relation between the GaN NWs and the individual grains of polycrystalline Ti foil [11]. Calabrese et al. studied the role of Ti foil surface condition on the formation of GaN NW in detail and found that the as-received rough Ti foil produced a tilted sparse GaN NW growth whereas vertically-aligned dense GaN NW ensemble was obtained once the substrate information was hindered upon Ti foil nitridation at 1000°C [13]. In the current study, the nitridation of Ti foil was performed at 700°C that can partially convert the foil surface resulting in the sparse growth of GaN NRs. The GaN NRs consists of two features: the formation of probe-

shaped neck with a height of ~ 50 – 70 nm close to the GaN/Ti interface and the elongated rod of length in the range of 150–280 nm having a nearly uniform diameter with hexagonal cross-sectional top [2,15,16] as we can see in the high magnification image in Fig. 3(d). The density and size distribution of GaN NRs grown at 700°C was statistically analyzed using several FESEM images. Inset of Fig. 3(d) represents the cross-sectional image and it was found that the average length of GaN NRs is 260 nm (the length falls in the range of 200–350 nm). The lateral size of top hexagonal facet of NRs varies in the range of 70–100 nm (average ~ 80 nm). The density of the GaN NRs grown at 700°C is $\sim 9.6 \times 10^8\text{ cm}^{-2}$, which is lower than that of GaN NRs grown at 600 – 650°C [10^9 – 10^{10} cm^{-2}]. Compared with the previous reports on PA-MBE growth of GaN NWs on Ti metal foils at 730 – 800°C [11–13], we have achieved the GaN NR growth on Ti foil using LMBE technique at 700°C which is 30°C lower.

The possible growth mechanism of various GaN nanostructures on Ti metal foils can be understood in terms of the influence of growth temperature on ad-atom diffusion and re-evaporation under N-rich growth condition. The ad-atom surface diffusion strongly depends on the growth temperature and is enhanced by raising the growth temperature. In case of low temperature (550°C) growth, the growth mode is controlled by ad-atom surface diffusion and the entire polycrystalline Ti surface is mostly covered by 3D GaN islands as the ad-atom surface diffusion is low [17]. The formation of elongated GaN NRs is related with increase in Ga ad-atom surface diffusion and re-evaporation and change of nucleation phase by raising growth temperature. The high density GaN NR growth can lead to increase of degree of coalescence of the NRs during the low temperature growth at 600 – 650°C [16–18]. When the GaN growth temperature is increased to 700°C under the similar growth condition, a prominent re-evaporation can be expected along with increased Ga ad-atom diffusion on NR sidewalls [19]. The increase in growth temperature strongly reduced the coalescence process and subsequently the density of GaN decreases with growth temperature according to island nucleation theory [17]. The large variation of NR length and sparse growth are related with the nucleation of GaN at grain boundaries or kink site as well as barrier for ad-atoms when it jumps from one surface/grain to another. The diffusion-induced repulsion of neighboring NRs can be the one of the reasons for the well separated GaN NR formation on nitridated Ti metal foil at 700°C [20].

The formation of probe-shaped GaN NR morphology can be explained based on the growth model proposed by Consonni et al. [21] and Fernandez-Garrido et al. [22] which rely on the inherent growth properties of GaN. At the initial stage of NR growth, unstable 3D GaN nuclei are spontaneously formed and matured into spherical-shaped 3D islands with growth time, which is later transformed into NR-like morphology with (0 0 0–1) top surface and (1 0–1 0) side-walls. Here, a significant amount of Ga adatom from the substrate is collected by each NR which grows sparsely on Ti foil and the Ga adatom concentration on the top surface of NR exceeds the threshold value for the 2D growth of side-walls; thus, it leads to an enhanced lateral growth in the beginning stage. However, with the evolution of NR diameter, the Ga adatom density becomes lesser at the top facet than the threshold value for 2D growth and, at that stage, the diameter of the NR is determined [22]. The vertical growth along c-axis proceeds resulting in the elongation of NR with a nearly constant diameter as the Ga adatoms arriving on the side-walls reach the NR top facet due to surface diffusion, which is likely at higher growth temperature. Else, the diameter of the NR can simultaneously increase with an order of magnitude lower in growth rate compared to its c-axis growth [23]. In addition, the c-axial growth is more preferential due to the low nucleation energy for c-plane as compared with m-plane as well as the low Ga adatom diffusion barrier [24–25].

Fig. 4(a) presents the 2θ - ω scan XRD pattern of LMBE grown GaN on nitridated Ti metal foil at 700°C . Similar XRD patterns were obtained for the GaN grown at lower temperatures as well. It was seen that only GaN (0 0 0 2) and (0 0 0 4) planes are observed along with Ti (1 1 0), (1 0 3)

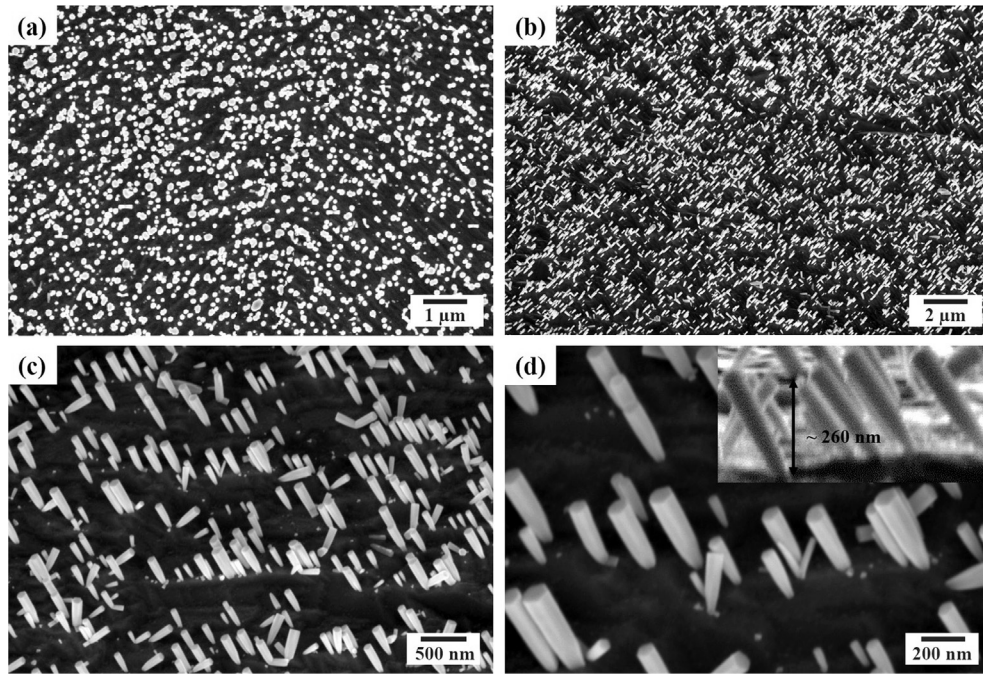


Fig. 3. FESEM images of GaN NRs grown on Ti foil at 700 °C temperature. (a) Plan-view; (b–d) 45° tilt-view FE-SEM images of different magnifications. Inset of (d) shows the cross-sectional view FE-SEM image.

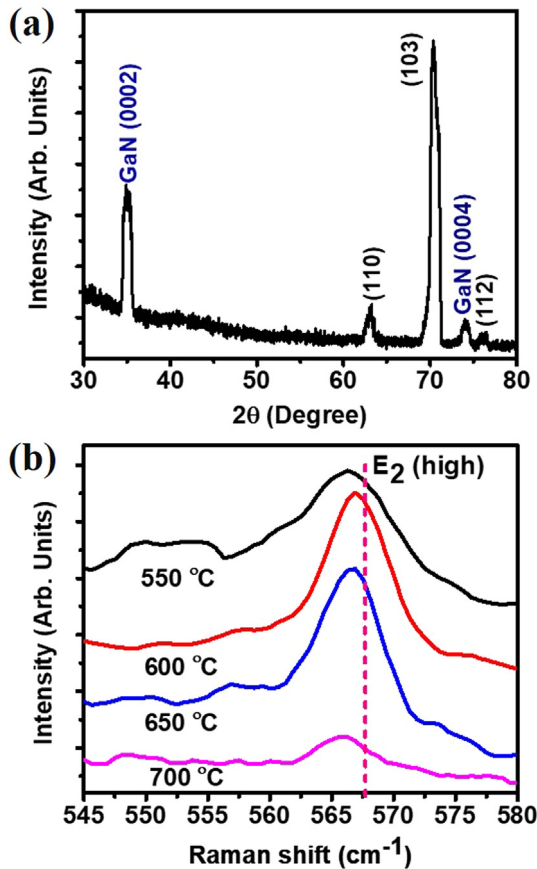


Fig. 4. (a) 2θ-ω XRD pattern of the LMBE grown GaN NRs on nitridated Ti metal foil at growth temperature of 700 °C. (b) Raman spectra of GaN grown on nitridated Ti foil at different temperature (550–700 °C); the dashed line represents the stress-free bulk GaN E₂ (high) position of 567.6 cm⁻¹.

and (1 1 2) peaks. This observation reveals that the GaN nanostructures have wurtzite crystalline structure and grow along the c-axis on Ti foil. Along with XRD, Raman spectroscopy measurements on GaN samples were performed at room temperature to identify the crystalline phase and estimation of biaxial stress present in LMBE grown GaN nanostructures. Fig. 4(b) shows the Raman spectra of GaN grown on Ti metal foil at temperature ranging from 550 to 700 °C. Here, the prominent GaN E₂ (high) phonon peak has been clearly observed for all the GaN samples that indicates the growth of wurtzite GaN phase on nitridated Ti foil and compliments the XRD study.

Further, the stress presented in the LMBE grown GaN samples has been analyzed from the Raman spectra shown in Fig. 4(b). The GaN E₂ (high) phonon mode is highly sensitive to the biaxial stress. GaN E₂ (high) peak shift towards lower side discloses the tensile stress behavior whereas blue shift represents the compressive stress with respect to stress-free GaN E₂ (high) peak position of 567.6 cm⁻¹ [26–28]. The E₂ (high) peak positions of LMBE grown GaN samples 566.33, 566.93, 566.66 and 566 cm⁻¹ for GaN growth at 550, 600, 650 and 700 °C are, respectively (Table 1). Here, the E₂ (high) peak position of all samples was shifted towards lower wavenumbers [Fig. 4(b)] as compared to stress-free GaN E₂(high). The biaxial stress (σ) present in GaN nanostructures is estimated using the following equation [27,28]:

$$\sigma = \frac{\omega - \omega_0}{4.3} \quad (1)$$

Table 1
Room temperature Raman and PL spectroscopy data of LMBE grown GaN on nitridated Ti metal foil at different growth temperatures.

Growth temperature	Raman spectral data		PL spectral data	
	E ₂ (high) position (cm ⁻¹)	E ₂ (high) FWHM (cm ⁻¹)	NBE peak position (eV)	NBE-FWHM (meV)
550 °C	566.33	13.99	3.38	260
600 °C	566.93	5.67	3.41	220
650 °C	566.66	5.98	3.41	205
700 °C	566.0	5.39	3.42	100

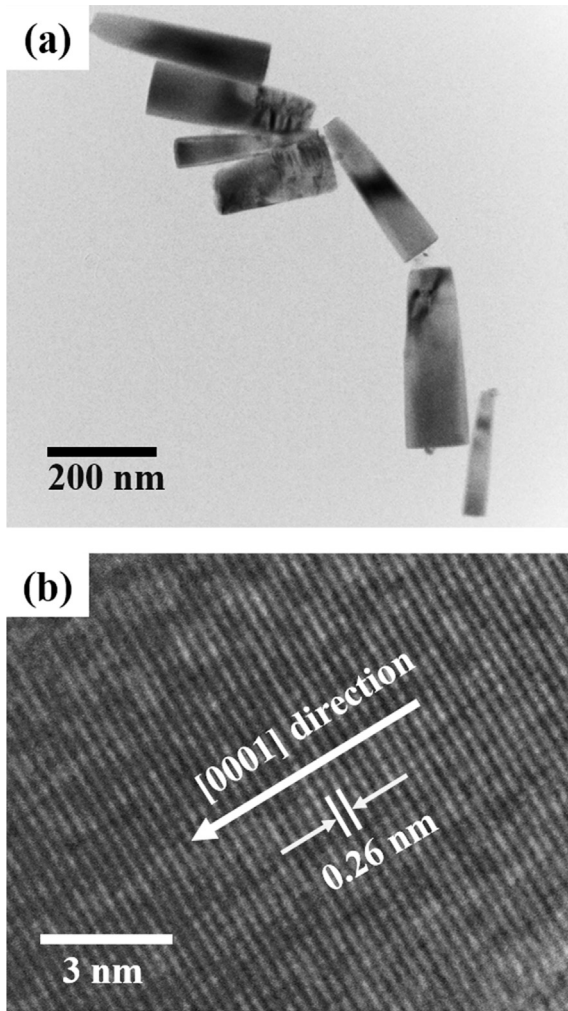


Fig. 5. (a) Bright-field TEM and (b) HR-TEM micrographs of GaN NRs grown on nitridated Ti metal foil at 700 °C temperature.

where ω and ω_0 are the E_2 (high) peak position of grown GaN and the E_2 (high) peak position of stress free GaN, respectively. The GaN E_2 (high) phonon peak shift reveals that the LMBE grown GaN NRs are under tensile biaxial stress. The estimated tensile biaxial stress of sparse GaN NRs on Ti foil grown at 700 °C showed a slightly high stress of 0.37 GPa as compared with low temperature grown dense NRs (0.15–0.22 GPa).

Fig. 5(a and b) shows the bright-field TEM micrographs taken on the GaN NRs dispersed over Cu grid. The TEM image revealed the growth of probe-shaped GaN NRs at 700 °C as seen in Fig. 5(a), which is in agreement with the FE-SEM observation. The crystalline nature of GaN NRs has also been examined with HR-TEM analysis. The atomic arrangements are clearly seen in the image [Fig. 5(b)] without any stacking faults, which revealed that the GaN NR is single crystalline in nature. The measured lattice spacing is 0.26 nm that resembles the growth of GaN NRs along the c-axis direction [11–14].

Fig. 6(a) shows the room temperature PL spectra of GaN nanostructures grown on Ti foil at different growth temperatures. High intensity near band-edge (NBE) emission peaks are obtained around 363 nm for all GaN samples. The GaN NRs grown at 700 °C showed the highest NBE luminescence intensity even though the GaN NR density and the surface coverage over Ti metal foil are lesser as compared with the low temperature grown samples [Figs. 2 and 3]. The NBE luminescence peak intensity gradually decreased with decreasing GaN growth temperature from 700 to 550 °C under the similar PL measurement condition. Further, the NBE peak positions of GaN

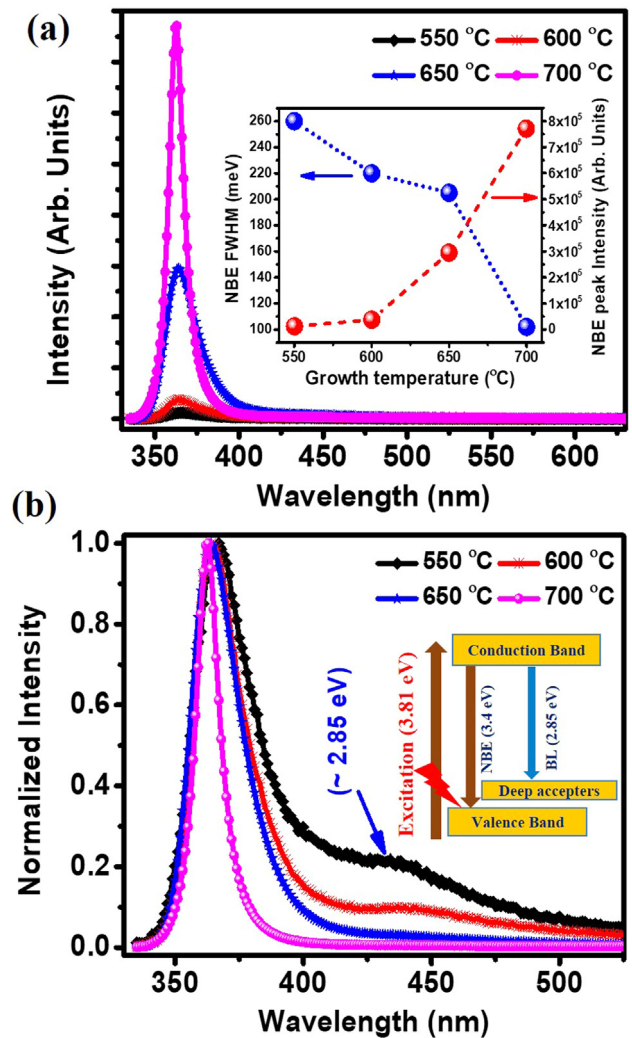


Fig. 6. (a) Room temperature PL spectrum of LMBE GaN nanostructures grown on Ti metal foil at different temperatures (550–700 °C); Inset shows the PL intensity and FWHM of NBE peak as a function of growth temperature. (b) The normalized PL spectra of GaN nanostructures; Inset shows the schematic band diagram of possible transition of optically excited electrons.

nanostructures grown at 550, 600, 650 and 700 °C are observed at 3.38, 3.41, 3.41 and 3.42 eV, respectively. The NBE peak position of LMBE grown GaN on Ti foil is close to that of bulk GaN (3.4 eV) [29]. The full-width at half maxima (FWHM) values of Lorentzian fitted NBE peaks are obtained as 260, 220, 205 and 100 meV corresponding to the GaN growth temperatures of 550, 600, 650 and 700 °C, respectively [Table 1]. The PL intensity and FWHM values of NBE peak are plotted against the growth temperature and the plot is shown as the inset of Fig. 6(a). The obtained FWHM values indicate that the optical quality of GaN nanostructures is enhanced by increasing the growth temperature. The GaN NRs grown at 700 °C shows a high optical quality of NBE peak with FWHM value of 100 meV at room temperature which is comparable to the NBE FWHM reported for GaN nanowires on Si(1 1 1) using metal-organic chemical vapor deposition [30].

The normalized PL spectra [Fig. 6(b)] of GaN samples show a prominent defect related blue luminescence (BL) shoulder peak at ~ 2.85 eV. The BL peak was suppressed by increasing GaN growth temperature and the NBE to BL peak intensity ratios are 4.7, 10 and 31 for the samples grown at 550, 600 and 650 °C, respectively. The BL peak intensity is negligible in case of GaN NRs grown at 700 °C. The origin of BL peak is related with the transitional excited electron from conduction band to unknown deep level acceptor defect state as denoted in the

inset of Fig. 6(b). However, there is no sign of yellow luminescence peak, which is the most commonly observed defect band for conventional GaN films grown on different substrates by various growth techniques [27–29]. These observations indicate that the sparse and regular array of GaN NRs grown at 700 °C on Ti foil possesses a good structural and optical quality.

4. Conclusions

The effect of growth temperature (550–700 °C) on the structural and optical properties of GaN nanostructures on nitridated Ti metal foil in LMBE growth process has been systematically studied. The FESEM studies revealed the formation of discrete and probe-shaped GaN NRs with an average diameter (length) of 80 (260) nm at growth temperature of 700 °C. Raman spectroscopy showed that the LMBE grown GaN NRs possess a nearly stress free wurtzite crystal structure. The HR-TEM study disclosed that the GaN NRs are grown along the c-axis on Ti metal foil and possess single crystalline property over the entire NR. The optical quality is improved with the increase of growth temperature and a low NBE FWHM of 100 meV has been obtained for GaN NRs grown at 700 °C, as deduced by PL spectroscopy measurements. The sparse and aligned GaN NRs on metal foils have an advantage for the growth of InGaGaN/GaN multi-layer structures on the side face of GaN NRs as it can avoid the shadow effects, which ultimately increase the device active area and improve the device efficiency.

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